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CLAIMS

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(57)[Claim(s)]

[Claim 1]

An anode oxidation method electrolyzing a processed member as the anode in an electrolysis solution, dissolving application-of-pressure carbon dioxide in water of the specified quantity, and generating an oxide film in an anode oxidation method which generates an oxide film on the surface of said processed member by using aerated water of acid concentration of pH 3-4 as an electrolysis solution.

[Claim 2]

The anode oxidation method according to claim 1 to which air bubbles of said aerated water are contacted into oxygen of said processed member circumference, and said oxygen is moved.

[Claim 3]

The anode oxidation method according to claim 1 which makes heat of an electrolysis solution emit to the exterior with air bubbles of said aerated water.

[Claim 4]

The anode oxidation method according to claim 2 or 3 which agitates said aerated water and always generates air bubbles.

[Claim 5]

The anode oxidation method according to claim 1 which gets mixed up or processes simultaneously degreasing of said processed member, and oxide film generation.

[Claim 6]

The anode oxidation method according to claim 1 which generates said oxide film in sealing and pressurized space.

[Claim 7]

The anode oxidation method according to claim 6 which processes simultaneously said oxide film generation and sealing of this coat.

[Claim 8]

The anode oxidation method according to claim 1 which dissolves supercritical carbon dioxide in water and generates an oxide film by using aerated water of acid concentration of pH 3-4 as an electrolysis solution.

[Claim 9]

The anode oxidation method according to claim 1 which decompresses and drains said electrolysis solution after anodic oxide film generation.

[Claim 10]

The anode oxidation method according to claim 1 which decompresses and heats said electrolysis solution after anodic oxide film generation, separates into water and carbon dioxide, and discharges or reuses these.

[Claim 11]

In an anodizing device which has arranged a processed member to an electrolysis solution accommodated in a cell, made electrolysis possible by having used this processed member as the anode, and enabled generation of an oxide film on the surface of said processed member, An anodizing device having provided water and application-of-pressure carbon dioxide in said cell so that introduction was possible, having dissolved application-of-pressure carbon dioxide in said water, having provided aerated water of acid concentration of pH 3-4 so that generation was possible, and making an oxide film generable by using this aerated water as an electrolysis solution.

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[Translation done.]

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]

This invention is suitable for oxide film generation and electrolytic polishing of aluminum, for example, While being able to abandon use of the electrolysis solution of strong acid nature, being able to adopt aerated water as an electrolysis solution, being able to generate an oxide film cheaply and promptly and being able to attain improvement of generation and rationalization of waste water treatment, Without requiring special equipment, prevent the rise in heat of an electrolysis solution, and generating oxygen of the processed member circumference is eliminated, The stable oxide film generation and a good oxide film can be obtained, and, moreover, it is related with the anode oxidation method and its processing unit which enabled it to aim at rationalization of oxide film generation processing, and improvement in productivity using supercritical carbon dioxide.

[0002]

[Description of the Prior Art]

For example, what is called an alumite (registered trademark) method that generates the oxide film of given thickness artificially to the body surface of aluminum, Like JP,9-176892,A, the electrolysis solution of strong acid nature, such as sulfuric acid and oxalic acid, was accommodated in the cell, the processed material made from aluminum was accommodated in this electrolysis solution, this processed material was used as the anode, and the body surface of aluminum was made to generate an oxide film by oxidation reaction with said electrolysis solution.

[0003]

However, this conventional oxide film generating method required the electrolysis solution of strong acid nature, such as sulfuric acid and oxalic acid, and required facilities for drainage special to wastewater of an electrolysis solution, and it had the problem of forcing it the work under the harmful generation of gas while generation cost increased and the installation cost increased.

And when generating the oxide film of high hardness, in order to have to set the bath temperature of a cell as low temperature and to prevent the bath temperature rise by the heat dissipation at the time of oxide film growth, cooling equipment and its cooling down were required, generation cost and an installation cost increased further, and productivity was bad.

[0004]

On the other hand, said oxide film consists of a porous bulk layer and a barrier layer which consists of infinite form alumina (aluminum<sub>2</sub>O<sub>3</sub>). Among these, many detailed holes are formed in the bulk layer by the side of the surface, coloring becomes possible by infiltrating a color into this hole, sealing which blockades said hole is performed after coloring, and the corrosion resistance on the surface of an oxide film and antifouling property were made to improve.

[0005]

However, said sealing takes moving of an anodizing tub, a separate processing tub, and the processed material after anodization, The water which sent steam under pressure into said processing tub, or was accommodated in the processing tub was heated at about 100 \*\*, this took addition of sealers, such as nickel acetate, the installation cost increased and there was a problem that a process became complicated.

[0006]

[Problem(s) to be Solved by the Invention]

This invention solves such a problem, for example, is suitable for oxide film generation and electrolytic

polishing of aluminum, While being able to abandon use of the electrolysis solution of strong acid nature, being able to adopt aerated water as an electrolysis solution, being able to generate an oxide film cheaply and promptly and being able to attain improvement of generation and rationalization of waste water treatment, Without requiring special equipment, prevent the rise in heat of an electrolysis solution, and generating oxygen of the processed member circumference is eliminated, The stable oxide film generation and a good oxide film can be obtained, and it aims at moreover providing the anode oxidation method and its processing unit which enabled it to aim at rationalization of oxide film generation processing, and improvement in productivity using supercritical carbon dioxide.

[0007]

[Means for Solving the Problem]

In an anode oxidation method which an invention of claim 1 electrolyzes a processed member as the anode in an electrolysis solution, and generates an oxide film on the surface of said processed member, Dissolve application-of-pressure carbon dioxide in water of the specified quantity, and an oxide film is generated by using aerated water of acid concentration of pH 3-4 as an electrolysis solution, While abandoning use of an electrolysis solution of strong acid nature like conventional sulfuric acid or oxalic acid, using a cheap and safe electrolysis solution and being able to aim at reduction of generation cost, and an improvement of work environment, it can drain without requiring special waste-water-treatment equipment, and enables it to aim at reduction of an installation cost, and prevention of environmental pollution.

[0008]

An invention of claim 2 contacts air bubbles of said aerated water into oxygen of said processed member circumference, moves said oxygen, eliminates an adverse effect of oxide film generation by this oxygen, and enables it to obtain stable oxide film generation and a good oxide film.

Without making heat of an electrolysis solution emit to the exterior, and requiring a special cooling method like before, an invention of claim 3 prevents a rise in heat of an electrolysis solution accompanying growth of an oxide film, and enables it to obtain a good oxide film with air bubbles of said aerated water.

[0009]

An invention of claim 4 agitates said aerated water, and always generates air bubbles, and while it eliminates oxygen generated around a processed member, he is trying to prevent a rise in heat of an electrolysis solution accompanying growth of an oxide film.

an invention of claim 5 — degreasing of said processed member, and oxide film generation — phase order — or it processes simultaneously, a part and an oxide film generation process of a head end process are performed rationally, and it enables it to aim at improvement in productivity

[0010]

While it prevents an outflow of carbon dioxide as an invention of claim 6 generates said oxide film in sealing and pressurized space, and it attains the recovery and reproductive facilitating, he is trying to urge improvement in acid concentration of aerated water to it.

An invention of claim 7 processes simultaneously said oxide film generation and sealing of this coat, rationalizes these down stream processing, and he is trying to cancel unreasonableness which requires a processing tub and incidental facilities, respectively as work which performs these processings independently is complicated.

[0011]

An invention of claim 8 dissolves supercritical carbon dioxide in water, and generates an oxide film by using aerated water of acid concentration of pH 3-4 as an electrolysis solution, He enables processing of degreasing, oxide film generation and sealing of a processed member, and washing processing by a single processing tub, and is trying to aim at improvement in the rationalization and productivity.

Without falling acid concentration of an electrolysis solution after use, as said electrolysis solution is decompressed and it drains after anodic oxide film generation, and requiring special waste-water-treatment equipment, an invention of claim 9 realizes easy and safe wastewater, and he is trying to prevent environmental pollution simultaneously.

[0012]

As said electrolysis solution after anodic oxide film generation is decompressed and heated, it separates into water and carbon dioxide and an invention of claim 10 discharges or reuses these, while it realizes easy and safe wastewater of said electrolysis solution, he is trying to aim at water after separation, and effective use of carbon dioxide.

[0013]

In an anodizing device which an invention of claim 11 has arranged a processed member to an electrolysis solution accommodated in a cell, enabled electrolysis of it by having used this processed member as the

anode, and enabled generation of an oxide film on the surface of said processed member, Provide water and application-of-pressure carbon dioxide in said cell so that introduction is possible, and application-of-pressure carbon dioxide is dissolved in said water, Provide aerated water of acid concentration of pH 3-4 so that generation is possible, and an oxide film is made generable by using this aerated water as an electrolysis solution, While abandoning use of an electrolysis solution of strong acid nature like conventional sulfuric acid or oxalic acid, using a cheap and safe electrolysis solution and being able to aim at reduction of generation cost, and an improvement of work environment, it can drain without requiring special waste-water-treatment equipment, and enables it to aim at reduction of an installation cost, and prevention of environmental pollution.

[0014]

[Embodiment of the Invention]

When the embodiment of the graphic display which applied this invention to the anode oxidation method of aluminum which is a processed member, or its alloy hereafter is described, in drawing 1, 1 is a cell of the owner bottom made from stainless steel, The inner surface is lined by vinyl chloride etc., and the opening of the upper part is easily equipped with the lid 2 removable.

[0015]

Into said cell 1, via the hooks 5 and 6, the processed member 3 made from aluminum which is a candidate for anodic oxide film generation, and the cathode materials 4, such as a lead plate, are hung so that receipts and payments are possible, and the anode and the negative pole of the electric power unit are connected to them.

The water 7 which is the generator material of an electrolysis solution, such as tap water and pure water, is accommodated in said organ bath 1, and the service pipe 9 which is open for free passage to the top peripheral surface at the water supply source 8 is connected.

[0016]

Among the figure, the opening and closing valve which inserted in the service pipe 9 the stirring bars 11, such as a stirrer which accommodated ten in the pars basilaris ossis occipitalis of the cell 1, and 12 are the heaters with which the peripheral surface of the cell 1 was equipped, and heating of said water 7 at 30-40 \*\* is enabled by prescribed temperature and an embodiment. In this case, the warm water warmed to said temperature may be supplied to the cell 1.

[0017]

The gas bottle 13 which is the application-of-pressure fluid or application-of-pressure gas which was safe and was stabilized as generator material of an electrolysis solution and which accommodated carbon dioxide is installed in the exterior of said cell 1, and the gas lead pipe 14 is connected to the lower peripheral surface of the cell 1 via the press pump 15 and the opening and closing valve 16.

[0018]

Said press pump 15 enables application of pressure of carbon dioxide for said carbon dioxide from more than atmospheric pressure by predetermined pressure and an embodiment than subcritical or supercritical pressure (7.4MPa), Said carbon dioxide is supplied in the cell 1, and said water 7 is dissolved in this, and the carbonic acid ( $H_2CO_3$ ) water which is an electrolysis solution is made generable.

[0019]

The communicating tube 17 was connected to the lower part of said cell 1, the opening and closing valve 18 was inserted in this pipe 17, and the downstream end is connected to the storage tank 19.

Said storage tank 19 is substantially [ as said cell 1 ] the same, and it was constituted by the approximately said capacity, the peripheral surface is equipped with the heater 20, and it is enabling heating of the reservoir liquid 21 accommodated in said tank 19 to prescribed temperature.

In the embodiment, the reservoir liquid 21 is heated at abbreviated 50 \*\*, and decomposition of the aerated water which is the main ingredients of this reservoir liquid 21 to water and carbon dioxide is enabled.

[0020]

The return pipes 22 and 23 are connected to the up-and-down peripheral surface of said storage tank 19, it is connected to said cell 1 and the press pump 15, and those other ends are enabling flowing back of said decomposed water and carbon dioxide at these.

The opening and closing valve which 24 and 25 inserted in said return pipes 22 and 23, the filter which 26 inserted in the return pipes 22 and 23 or ion-exchange resin, and 27 and 28 are the air vent holes formed in the lids 2 and 2 among a figure.

[0021]

The exhaust pipe 29 is connected to the lower part of said storage tank 19, the downstream end is open for free passage in sewerage, and 30 is the opening and closing valve inserted in said exhaust pipe 29.

[0022]

The anode oxidation method constituted in this way and its processing unit, The storage tank 19 which can be accommodated is primarily required for the cell 1 which can be opened and closed, the water supply source 8 which can supply the water 7 to this cell 1, the gas bottle 13 which can supply liquid carbon dioxide high-density in a fluid or the shape of a gas, and an embodiment to said cell 1, and the treating solution after said anodic oxide film generation processing.

[0023]

That is, use of the electrolysis solution of the strong acid nature by conventional sulfuric acid, oxalic acid, etc. is abandoned, and generation cost can be reduced by using the cheap and safe water 7 and carbon dioxide, and the work environment under the harmful generation of gas is improved, and the safety of work can be planned.

And special facilities for drainage like the conventional neutralization equipment are not taken to abandon use of the electrolysis solution of strong acid nature, but reduction of an installation cost can be aimed at by it.

[0024]

And by always agitating the electrolysis solution of aerated water via the stirring bar 10, it is generated in large quantities, and by movement of these air bubbles, air bubbles detailed to an electrolysis solution stimulate heat dissipation of bath temperature, and prevent that temperature up.

Therefore, while oxide film generation is stabilized and a good oxide film can be obtained by preventing the rise in heat of the electrolysis solution accompanying growth of an oxide film, for example, and maintaining that fixed temperature state, the cooling method for it is omitted or that small capability-ization can be attained.

[0025]

It is decomposed into water and carbon dioxide by the storage tank 19 like the after-mentioned, and since the treating solution after anodizing is flowing back and reusing them to the cell 1 and the press pump 15, it can aim at reduction of those effective use and consumption.

[0026]

Next, when carrying out anodizing of the processed member 3 with said processing unit, the processed member 3 is pretreated beforehand, the processed member 3 after degreasing and etching, chemical polishing, electrolytic polishing, or satin finish is accommodated in the cell 1, and this is connected to the anode of an electric power unit.

Then, it equips with the lid 2, the water 7 is supplied to the cell 1 from the water supply source 8, and said processed member 3 is made immersed into this water 7.

[0027]

Carbon dioxide is supplied for said water 7 to the cell 1 from the gas bottle 13 after constant feeding, and this is pressurized by predetermined pressure and an embodiment with the press pump 15 more than atmospheric pressure, and also said water 7 is heated at 30-40 \*\* via the heater 12.

It gets mixed up with this, the stirring bar 10 is operated, the electrolysis solution 7 is agitated, and entropy of the temperature distribution and acid concentration distribution is carried out.

[0028]

If it does in this way, underwater [ of the cell 1 / 7 ] is conjointly gone up flourishing in the state of bubbling with churning of the stirring bar 10, and said carbon dioxide will dissolve in this water 7, will generate carbonic acid ( $\text{H}_2\text{CO}_3$ ), and will acidify said water 7.

In this case, since it is pressurized more than atmospheric pressure, and said water 7 is heated and the dissolution of carbon dioxide is urged to said carbon dioxide, the acid concentration of said water 7 rises and it forms promptly acid (PHs 3-4) sufficient concentration for oxide film generation. Since the acid concentration of said aerated water falls temporally with use, it sends in carbon dioxide timely and maintains acid predetermined concentration.

[0029]

If right current is energized to the processed member 3 under such a situation, the processed member 3 will be oxidized with said acidified electrolysis solution, and the anodic oxide film of infinite form alumina ( $\text{aluminum}_2\text{O}_3$ ) will be formed in the body surface.

[0030]

In that case, oxygen occurs to the circumference of the processed member 3 by the electrolysis of water,

and there is \*\*\*\* which checks generation of an anodic oxide film in it. However, it collides or contacts and said oxygen is moved to the carbon dioxide of the shape of said bubbling, and since said oxidation reaction is maintained, an anodic oxide film grows smoothly and promptly.

[0031]

On the other hand, with growth of such an oxide film, the aerated water which is an electrolysis solution generates heat, and there is \*\*\*\* which the temperature rises and checks generation of a good anodic oxide film.

However, in an embodiment, since detailed air bubbles are generated in large quantities, stimulate heat dissipation of bath temperature by movement of these air bubbles and prevent that temperature up, the stable oxide film generation and a good oxide film can be obtained.

Since the cooling method for it is omitted or that small capability-ization can be attained in that case, reduction of that part installation cost can be aimed at.

[0032]

And predetermined time execution of said anodizing is carried out, supply of carbon dioxide is suspended, the drive of the stirring bar 10 is suspended, and the opening and closing valve 18 is opened in the place which was able to obtain the anodic oxide film of sufficient thickness.

[0033]

If it does in this way, the inside of the cell 1 is decompressed and the solubility of said NI carbon monoxide falls, and the treating solution will be led to the communicating tube 17, it will extrude to the storage tank 19, and the opening and closing valve 18 will be closed in the place which the whole quantity moved to the storage tank 19.

[0034]

For this reason, since the reservoir liquid 21 in said storage tank 19 is decompressed and the solubility of NI carbon monoxide falls, that acid concentration falls quickly and \*\*\*\* of the actual harm to environment is lost. Then, the opening and closing valve 28 can be opened and said reservoir liquid 21 can be discharged from the exhaust pipe 27 as it is to sewerage.

[0035]

In that case, in the reservoir liquid 19 by for example, the thing which NI carbon monoxide disappears out of said reservoir liquid 21 when a heavy metal exists, therefore it dissociates from aerated water and precipitates in said tank 19, the filter (graphic display abbreviation) formed in the exhaust pipe 29 is passed — while becoming callable together with other foreign matters and oxide films, securing the safety of said wastewater and preventing environmental pollution, after the recovery can be processed as usual waste.

[0036]

On the other hand, this invention can reuse said reservoir liquid 21, heats the heater 20 in that case, and heats the reservoir liquid 21 in the storage tank 19 at abbreviated 50 \*\*.

If it does in this way, the aerated water of the reservoir liquid 21 will be divided into carbon dioxide and water, and these will be divided into a vapor-liquid bilayer. That is, gas-like carbon dioxide is located in a higher rank, and water is located in a low rank.

[0037]

Then, if the opening and closing valves 25 and 26 are opened, said disassembled carbon dioxide and water will be led to the return pipes 22 and 23, and will move to the cell 1 and the press pump 15, and those reuse will be attained.

In that case, said carbon dioxide and water are removed in a heavy metal, an oxide film, and a foreign matter by the filters 26 and 26 inserted in each return pipes 22 and 23.

In this case, since carbon dioxide falls out from the reservoir liquid 21 thoroughly and comes out of it by said decomposition, said heavy metal, an oxide film, etc. precipitate thoroughly, and these can be collected with sufficient accuracy.

[0038]

Thus, while this invention is a cheap raw material of water and carbon dioxide, generating the anodic oxide film of the processed member 3, abandoning use of the electrolysis solution of the strong acid nature by conventional sulfuric acid, oxalic acid, etc. and reducing generation cost, The work environment under the harmful generation of gas is improved, the safety of work can be planned, special facilities for drainage like the conventional neutralization equipment are not taken to abandon use of the electrolysis solution of strong acid nature moreover, but reduction of an installation cost can be aimed at by it.

[0039]

While detailed air bubbles are generated to the electrolysis solution of aerated water in large quantities,

urging heat dissipation of bath temperature to it by movement of these air bubbles, preventing that temperature up, preventing the rise of the bath temperature at the time of anodic oxide film growth and being able to aim at generation of a good anodic oxide film, the cooling method for it is omitted or that small capability-ization can be attained.

[0040]

The storage tank 19 separates into water and carbon dioxide, and the treating solution after anodizing flows back and reuses them to the cell 1 and the press pump 15, and aims at reduction of those effective use and consumption.

[0041]

Although the processed member 3 pretreated beforehand is accommodated in the cell 1 in the above-mentioned embodiment, without carrying out the processing concerned beforehand, it accommodates in the cell 1 and degreasing treatment can be processed simultaneously.

That is, the pressurized carbon dioxide is sent into the water 7 in the cell 1 after accommodating the processed member 3 in the cell 1, high speed movement of this is microatomized and carried out, it exfoliates and the oil and fat content adhering to the surface of the processed member 3 is made to degrease by making it collide with the processed member 3.

[0042]

In this case, since said carbon dioxide is supplied from the lower part of the cell 1 and this goes up underwater [ 7 ] in the state of bubbling, while said carbon dioxide dissolves in the water 7 promptly, being saturated and urging the rise of solubility, A stirring effect conjointly as uniform as said stirring bar 10 and precise is obtained, and said degreasing operation is increased.

[0043]

If said water 7 is sprayed in the cell 1 at misty state, said carbon dioxide is simultaneously supplied instead of said method and they are mixed, while those touch areas will increase further and urging the rise of solubility, a precise stirring effect is obtained and said degreasing operation is increased further.

[0044]

Drawing 2 and drawing 3 show other embodiments of this invention, and use the same numerals for the composition of the above-mentioned embodiment, and a corresponding portion.

Among these, drawing 2 shows a 2nd embodiment of this invention, this embodiment installs the electrolysis solution generation machine 31 in the outside of the cell 1, and connects said gas lead pipe 14 and the service pipe 9 to this generation machine 31, and the carbon dioxide and water which were introduced into this generation machine 31 are reacted, The aerated water 32 which is an electrolysis solution is generated, and this is supplied to the cell 1 via the lead pipe 33.

[0045]

It is the opening and closing valve which 34 inserted in the lead pipe 33, and the exhaust valve which 35 attached to the lid 2 among a figure, it opens by the detection operation of the surface sensor 36 provided in the upper part of the cell 1, and while discharging to the exterior the carbon dioxide which stagnated between the electrolysis solution 32 and the lid 2, prevention of the extravasation of the electrolysis solution 32 is enabled.

[0046]

That is, this embodiment generates the aerated water which is an electrolysis solution with the external electrolysis solution generation machine 31, and he is trying to attain facilitating of aerated water generation, and miniaturization and cheap-izing of aerated water generation equipment by supplying this to the cell 1.

Said lid 2 is constituted in crookedness and elastic bellows shape, via this lid 2, it leaves a part and the opening of the cell 1 is blockaded, and the tip part is engrossed into the electrolysis solution 32, and most openings of the cell 1 are blockaded.

[0047]

And an anodic oxide film generate time makes the cell 1 a half-sealed state like a graphic display, controls the outflow of the carbon dioxide which stagnated between the electrolysis solution 32 and the lid 2 as much as possible, discharges the carbon dioxide which stagnated more than fixed with said exhaust valve 35, and is aiming at extravasation prevention of the safety of work and the electrolysis solution 32.

In this case, if the carbon dioxide which stagnated in the upper part of the cell 1 is flowed back to said electrolysis solution generation machine 31, that effective use can be aimed at.

If carbon dioxide is directly sent into the cell 1 separately from the gas lead pipe 14, the acid concentration of the electrolysis solution 32 can be maintained uniformly.

[0048]

This real gestalt is constituted by drawing 3 showing a 3rd embodiment of this invention in the resisting pressure structure which can seal the cell 1 and the storage tank 19, among these carbon dioxide and the water 7 are introduced into the cell 1 uniquely or simultaneous, and formation of them to a supercritical state is enabled.

[0049]

The storage tank 19 stores the treating fluid of the cell 1 primarily, and carries out vapor liquid separation of that treating fluid, is reproduced, and this water and carbon dioxide that were reproduced are refluxed to the cell 1 and the press pump 15, and it is enabling reuse of them.

[0050]

The processed member 3 before degreasing to said cell 1 is accommodated, after sealing this tub 1, carbon dioxide is introduced, this carbon dioxide is formed in a supercritical state, i.e., 7.4MPa, and not less than 31 \*\* via the press pump 15 and the heater 12, and degreasing washing of the processed member 3 is carried out.

[0051]

Open the opening and closing valve 18 after said degreasing washing, and the carbon dioxide after washing is sent out to the storage tank 19, The water 7 and carbon dioxide of the specified quantity are introduced into the cell 1 after closing the opening and closing valve 18, these are dissolved, aerated water is generated, a predetermined surface-active agent is added to this, and the emulsion state of supercritical carbon dioxide is formed.

[0052]

In this case, since the inside of the cell 1 is a high pressure state, the solubility of the carbon dioxide to the water 7 rises so much, and the acid concentration of aerated water rises.

And while operating the stirring bar 10, agitating an electrolysis solution and making a lot of [ detailed and ] air bubbles generate in this electrolysis solution, Right current is energized to the processed member 3, said processed member 3 which carried out degreasing treatment is oxidized with an electrolysis solution, and the anodic oxide film of infinite form alumina ( $\text{aluminum}_2\text{O}_3$ ) is generated by the body surface.

[0053]

Energization is stopped after predetermined time processing, and the opening and closing valve 18 is opened, and the water 7 and carbon dioxide which were divided into the vapor-liquid bilayer are sent out to the storage tank 19.

While a fixed flow occurs and this washes the processed member 3 in the system of the cell 1 in that case, the desiccation is urged and washing in cold water after the conventional anodizing can be excluded.

[0054]

In this way, about the generated anodic oxide film, when the artificer tried coloring, coloring was impossible.

Since the inside of the cell 1 is put on an anodic oxide film generate time by the high pressure state of 7.4MPa, sealing is performed simultaneously with anodic oxide film generation, and this is considered to be the result of plugging up the hole of the bulk layer of an anodic oxide film.

[0055]

Therefore, if an anodic oxide film is generated under supercritical carbon dioxide, since sealing is performed simultaneously, there is no necessity for complicated moving of the processed member 3 to the treating layer and this treating layer for sealing like before, and reduction of an installation cost, miniaturization of equipment, and improvement in productivity can be aimed at.

[0056]

Although each above-mentioned embodiment has applied this invention to anodic oxide film generation, it is possible not only this but to apply to electrolytic polishing of the substantially same principle as an anode oxidation method.

[0057]

[Effect of the Invention]

As mentioned above, the invention of claim 1 dissolves application-of-pressure carbon dioxide in the water of the specified quantity, Since the oxide film was generated by using the aerated water of the acid concentration of pH 3-4 as an electrolysis solution, abandon use of the electrolysis solution of strong acid nature like conventional sulfuric acid or oxalic acid, and a cheap and safe electrolysis solution is used, While aiming at reduction of generation cost, and an improvement of work environment, it can drain without requiring special waste-water-treatment equipment, and reduction of an installation cost and



prevention of environmental pollution can be aimed at.

[0058]

Since the invention of claim 2 contacted the air bubbles of said aerated water into oxygen of said processed member circumference and moved said oxygen, it can eliminate the adverse effect of the oxide film generation by this oxygen, and can obtain the stable oxide film generation and a good oxide film. With the air bubbles of said aerated water, without requiring a special cooling method like before, since the heat of the electrolysis solution was made to emit to the exterior, the invention of claim 3 can prevent the rise in heat of the electrolysis solution accompanying growth of an oxide film, and can obtain a good oxide film.

[0059]

Discharge of the heat of the electrolysis solution accompanying growth of an oxide film is urged to it, it prevents the rise in heat of an electrolysis solution, and is effective in the ability to obtain a good oxide film while it eliminates the oxygen generated around a processed member and can obtain the stable oxide film generation, since the invention of claim 4 agitated said aerated water and always generated air bubbles.

the invention of claim 5 — degreasing of said processed member, and oxide film generation — phase order — or since it processed simultaneously, the part and oxide film generation process of a head end process can be performed rationally, and improvement in productivity can be aimed at.

[0060]

Improvement in the acid concentration of aerated water can be urged to it while it prevents the outflow of carbon dioxide and can attain the recovery and reproductive facilitating, since the invention of claim 6 generated said oxide film in sealing and pressurized space.

Since the invention of claim 7 processed simultaneously said oxide film generation and sealing of this coat, it can rationalize these down stream processing and can cancel the unreasonableness which requires a processing tub and incidental facilities, respectively as the work which performs these processings independently is complicated.

[0061]

Since the invention of claim 8 dissolves supercritical carbon dioxide in water, uses the aerated water of the acid concentration of pH 3-4 as an electrolysis solution and the oxide film was generated, Degreasing, oxide film generation and sealing of a processed member, and washing processing can be processed by a single processing tub, and improvement in the rationalization and productivity can be aimed at.

[0062]

After anodic oxide film generation, without falling the acid concentration of the electrolysis solution after use, and requiring special waste-water-treatment equipment, since said electrolysis solution is decompressed and it was made to drain, the invention of claim 9 can realize easy and safe wastewater, and can prevent environmental pollution simultaneously.

It can aim at the water after separation, and effective use of carbon dioxide while it can realize easy and safe wastewater of said electrolysis solution, since said electrolysis solution after anodic oxide film generation is decompressed and heated, it separates into water and carbon dioxide and the invention of claim 10 discharged or reused these.

[0063]

The invention of claim 11 provides water and application-of-pressure carbon dioxide in a cell so that introduction is possible, and it dissolves application-of-pressure carbon dioxide in said water, Since the aerated water of the acid concentration of pH 3-4 was provided so that generation was possible, and the oxide film was made generable by using this aerated water as an electrolysis solution, abandon use of the electrolysis solution of strong acid nature like conventional sulfuric acid or oxalic acid, and a cheap and safe electrolysis solution is used, While being able to aim at reduction of generation cost, and an improvement of work environment, it can drain without requiring special waste-water-treatment equipment, and reduction of an installation cost and prevention of environmental pollution can be aimed at.

[Brief Description of the Drawings]

[Drawing 1] It is an explanatory view showing the embodiment which applied this invention to anodizing of the aluminum product.

[Drawing 2] In the explanatory view showing a 2nd embodiment of this invention, an electrolysis solution is generated in the exterior of a cell and this is supplied to the cell.

[Drawing 3] In the explanatory view showing a 3rd embodiment of this invention, supercritical carbon dioxide is introduced into the cell pressure-proofed and sealed, and anodizing of the water is dissolved and

carried out to this.

[Description of Notations]

1 Cell

3 Processed member

7 Water

.....  
[Translation done.]

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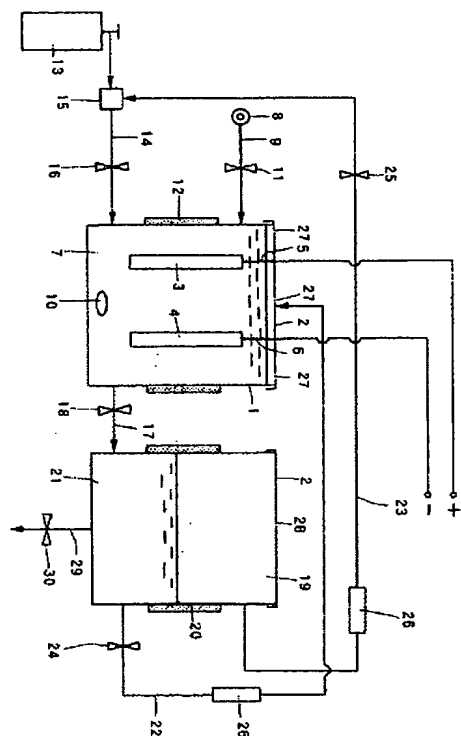
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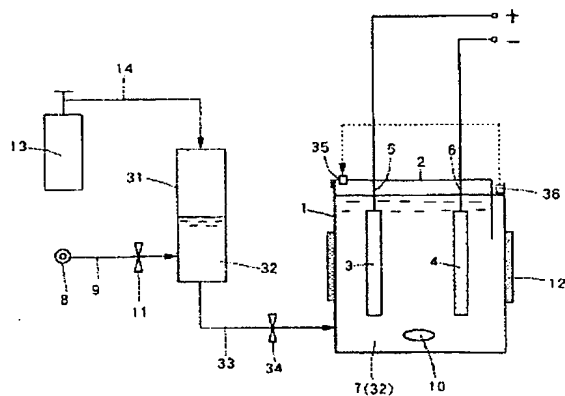
3.In the drawings, any words are not translated.

## DRAWINGS

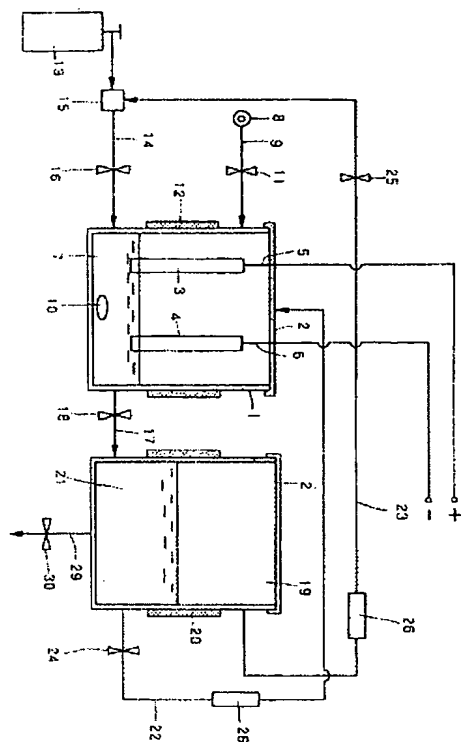
[Drawing 1]



[Drawing 2]



[Drawing 3]



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(21) 出願番号	特願2001-387780 (P2001-387780)	(73) 特許権者	500398289
(22) 出願日	平成13年12月20日 (2001.12.20)		吉田 英夫
(65) 公開番号	特開2003-183888 (P2003-183888A)		埼玉県所沢市松が丘2-9-2
(43) 公開日	平成15年7月3日 (2003.7.3)	(74) 代理人	100085110
審査請求日	平成15年5月23日 (2003.5.23)		弁理士 千明 武
		(72) 発明者	吉田 英夫
			東京都東村山市久米川町5-33-6
		(72) 発明者	阿部 健太郎
			東京都東村山市久米川町5-33-6
			株式会社 ワイビー
			システム 内
		(72) 発明者	曾根 正人
			東京都小金井市本町3-11-8
		審査官	瀧口 博史

最終頁に続く

(54) 【発明の名称】 陽極酸化法およびその処理装置

(57) 【特許請求の範囲】

【請求項 1】

電解液中で被処理部材を陽極として電解し、前記被処理部材の表面に酸化皮膜を生成する陽極酸化法において、所定量の水に加圧二酸化炭素を溶解し、pH 3 ~ 4の酸性濃度の炭酸水を電解液として酸化皮膜を生成することを特徴とする陽極酸化法。

【請求項 2】

前記被処理部材周辺の酸素に前記炭酸水の気泡を接触させて、前記酸素を移動させる請求項 1 記載の陽極酸化法。

【請求項 3】

前記炭酸水の気泡により、電解液の熱を外部へ放出させる請求項 1 記載の陽極酸化法。

【請求項 4】

前記炭酸水を攪拌して常時気泡を生成する請求項 2 または請求項 3 記載の陽極酸化法。

【請求項 5】

前記被処理部材の脱脂と酸化皮膜生成を、相前後し若しくは同時に処理する請求項 1 記載の陽極酸化法。

【請求項 6】

密閉かつ加圧空間で前記酸化皮膜を生成する請求項 1 記載の陽極酸化法。

【請求項 7】

前記酸化皮膜生成と該皮膜の封孔処理とを同時に処理する請求項 6 記載の陽極酸化法。

【請求項 8】

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皮膜を生成でき、生成作業の改善と排水処理の合理化を図れるとともに、特別な設備を要することなく、電解液の温度上昇を防止し、また被処理部材周辺の発生酸素を排除して、安定した酸化皮膜生成と良質な酸化皮膜を得られ、しかも超臨界二酸化炭素を用いて、酸化皮膜生成処理の合理化と生産性の向上を図れるようにした、陽極酸化法およびその処理装置を提供することを目的とする。

【0007】

【課題を解決するための手段】

請求項1の発明は、電解液中で被処理部材を陽極として電解し、前記被処理部材の表面に酸化皮膜を生成する陽極酸化法において、所定量の水に加圧二酸化炭素を溶解し、pH 3～4の酸性濃度の炭酸水を電解液として酸化皮膜を生成するようにして、従来の硫酸や

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リン酸のような強酸性の電解液の使用を廃し、安価かつ安全な電解液を使用して、生成コストの低減と作業環境の改善を図れるとともに、特別な排水処理設備を要することなく排水でき、設備費の低減と環境汚染の防止を図れるようにしている。

【0008】

請求項2の発明は、前記被処理部材周辺の酸素に前記炭酸水の気泡を接触させて、前記酸素を移動させ、該酸素による酸化皮膜生成の悪影響を排除し、安定した酸化皮膜生成と良質な酸化皮膜を得られるようにしている。

請求項3の発明は、前記炭酸水の気泡により、電解液の熱を外部へ放出させ、従来のような特別な冷却手段を要することなく、酸化皮膜の成長に伴う電解液の温度上昇を防止し、良質な酸化皮膜を得られるようにしている。

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【0009】

請求項4の発明は、前記炭酸水を攪拌して常時気泡を生成し、被処理部材周辺に発生する酸素を排除するとともに、酸化皮膜の成長に伴う電解液の温度上昇を防止するようにしている。

請求項5の発明は、前記被処理部材の脱脂と酸化皮膜生成を、相前後若しくは同時に処理し、前処理工程の一部と酸化皮膜生成工程を合理的に行ない、生産性の向上を図れるようにしている。

【0010】

請求項6の発明は、密閉かつ加圧空間で前記酸化皮膜を生成するようにして、二酸化炭素の流出を防止し、その回収と再生の容易化を図るとともに、炭酸水の酸性濃度の向上を促すようにしている。

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請求項7の発明は、前記酸化皮膜生成と該皮膜の封孔処理とを同時に処理し、これらの処理工程を合理化して、これらの処理を別々に行なう作業の煩雑と、処理槽および付帯設備をそれぞれ要する不合理と、を解消するようにしている。

【0011】

請求項8の発明は、超臨界二酸化炭素を水に溶解し、pH 3～4の酸性濃度の炭酸水を電解液として酸化皮膜を生成するようにして、被処理部材の脱脂と酸化皮膜生成、および封孔処理と洗浄処理とを単一の処理槽で処理可能にし、その合理化と生産性の向上を図るようにしている。

請求項9の発明は、陽極酸化皮膜生成後、前記電解液を減圧して排水するようにして、使用後の電解液の酸性濃度を低下し、特別な排水処理設備を要することなく、容易かつ安全な排水を実現し、同時に環境汚染を防止するようにしている。

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【0012】

請求項10の発明は、陽極酸化皮膜生成後の前記電解液を減圧かつ加熱して水と二酸化炭素に分離し、これらを排出または再利用するようにして、前記電解液の容易かつ安全な排水を実現するとともに、分離後の水と二酸化炭素の有効利用を図るようにしている。

【0013】

請求項11の発明は、電解槽に収容した電解液に被処理部材を配置し、該被処理部材を陽極として電解可能にし、前記被処理部材の表面に酸化皮膜を生成可能にした陽極酸化処理装置において、前記電解槽に水と加圧二酸化炭素とを導入可能に設け、前記水に加圧二

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酸化炭素を溶解して、pH 3～4の酸性濃度の炭酸水を生成可能に設け、該炭酸水を電解液として酸化皮膜を生成可能にして、従来の硫酸やリン酸のような強酸性の電解液の使用を廃し、安価かつ安全な電解液を使用して、生成コストの低減と作業環境の改善を図れるとともに、特別な排水処理設備を要することなく排水でき、設備費の低減と環境汚染の防止を図れるようにしている。

#### 【0014】

##### 【発明の実施の形態】

以下、本発明を被処理部材であるアルミニウム若しくはその合金の陽極酸化法に適用した図示の実施形態について説明すると、図1において1はステンレス鋼製の有底の電解槽で、その内面を塩化ビニール等でライニングしており、その上側の開口部に蓋体2が容易に

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着脱可能に装着されている。

#### 【0015】

前記電解槽1内に、陽極酸化皮膜生成対象であるアルミニウム製の被処理部材3と、鉛板等の陰極材料4とが、引掛け5、6を介して出し入れ可能に吊り下げられ、それらに電源装置の陽極と陰極が接続されている。

前記浴槽1内に電解液の生成素材である水道水、純水等の水7が収容され、その上部周面に給水源8に連通する給水管9が接続されている。

#### 【0016】

図中、10は電解槽1の底部に収容したスターラ等の攪拌子11は給水管9に介挿した開閉弁、12は電解槽1の周面に装着したヒータで、前記水7を所定温度、実施形態では30～40℃に加熱可能にしている。この場合、前記温度に加温した温水を電解槽1へ供給してもよい。

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#### 【0017】

前記電解槽1の外部に、電解液の生成素材として、安全で安定した加圧液体若しくは加圧気体である、例えば二酸化炭素を収容したガス容器13が設置され、そのガス導管14が圧縮ポンプ15および開閉弁16を介して、電解槽1の下部周面に接続されている。

#### 【0018】

前記圧縮ポンプ15は、前記二酸化炭素を所定圧、実施形態では二酸化炭素を大気圧以上から亜臨界若しくは超臨界圧(7.4MPa)以上に加圧可能にし、前記二酸化炭素を電解槽1内に供給し、かつこれに前記水7を溶解させて、電解液である炭酸(H<sub>2</sub>CO<sub>3</sub>)水を生成可能にしている。

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#### 【0019】

前記電解槽1の下部に連通管17が接続され、該管17に開閉弁18が介挿され、その下流側端部を貯留タンク19に接続している。

前記貯留タンク19は、前記電解槽1と実質的に同一かつ略同容積に構成され、その周面にヒータ20を装着していて、前記タンク19内に収容した貯留液21を所定温度に加熱可能にしている。

実施形態では貯留液21を略50℃に加熱し、該貯留液21の主成分である炭酸水を水と二酸化炭素に分解可能にしている。

#### 【0020】

前記貯留タンク19の上下周面にリターンパイプ22、23が接続され、それらの他端が前記電解槽1と圧縮ポンプ15に接続され、これらに前記分解した水と二酸化炭素を還流可能にしている。

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図中、24、25は前記リターンパイプ22、23に介挿した開閉弁、26はリターンパイプ22、23に介挿したフィルタ若しくはイオン交換樹脂、27、28は蓋体2、2に形成した抜気孔である。

#### 【0021】

前記貯留タンク19の下部に排出管29が接続され、その下流側端部が下水道に連通しており、30は前記排出管29に介挿した開閉弁である。

#### 【0022】

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このように構成した陽極酸化法およびその処理装置は、開閉可能な電解槽 1 と、該電解槽 1 に水 7 を供給可能な給水源 8 と、前記電解槽 1 に液体若しくは気体状、実施形態では密度の高い液体二酸化炭素を供給可能なガス容器 13 と、前記陽極酸化皮膜生成処理後の処理液を一次的に収容可能な貯留タンク 19 とを要する。

#### 【0023】

すなわち、従来の硫酸や硝酸等による強酸性の電解液の使用を廃し、安価かつ安全な水 7 と二酸化炭素を用いることで、生成コストを低減でき、また有害なガス発生下での作業環境を改善し、作業の安全性を図れる。

しかも、強酸性の電解液の使用を廃することで、従来の中和設備のような特別な排水設備を要せず、設備費の低減を図れる。

#### 【0024】

しかも、炭酸水の電解液を攪拌子 10 を介して常時攪拌することで、電解液に微細な気泡が大量に生成され、この気泡の移動によって浴温の放熱を促し、その昇温を防止する。

したがって、例えば酸化皮膜の成長に伴う電解液の温度上昇を防止し、その一定の温度状態を維持することで、酸化皮膜生成が安定し、良質な酸化皮膜を得られる一方、このための冷却手段を省略し、若しくはその小能力化を図れる。

#### 【0025】

更に、陽極酸化処理後の処理液は、後述のように貯留タンク 19 で水と二酸化炭素に分解され、それらを電解槽 1 および圧縮ポンプ 15 に還流して、再利用しているから、それらの有効利用と消費の節減を図れる。

#### 【0026】

次に、前記処理装置によって被処理部材 3 を陽極酸化処理する場合は、被処理部材 3 を予め前処理し、脱脂およびエッチング若しくは化学研磨または電解研磨、或いは梨地加工後の被処理部材 3 を電解槽 1 内に収容し、これを電源装置の陽極に接続する。

この後、蓋体 2 を装着し、給水源 8 から水 7 を電解槽 1 へ供給し、該水 7 の中に前記被処理部材 3 を浸漬させる。

#### 【0027】

前記水 7 を定量供給後、ガス容器 13 から二酸化炭素を電解槽 1 へ供給し、これを圧縮ポンプ 15 で所定圧、実施形態では大気圧以上に加圧し、更にヒータ 12 を介して前記水 7 を 30～40℃に加熱する。

また、これと前後して攪拌子 10 を作動し、電解液 7 を攪拌して、その温度分布と酸性濃度分布を一様化する。

#### 【0028】

このようにすると、前記二酸化炭素が攪拌子 10 の攪拌と相俟って、電解槽 1 の水中 7 をバブリング状態で旺盛に上昇し、該水 7 に溶解して炭酸 ( $\text{H}_2\text{CO}_3$ ) を生成し、前記水 7 を酸性化する。

この場合、前記二酸化炭素は大気圧以上に加圧され、また前記水 7 が加熱されて、二酸化炭素の溶解を促すから、前記水 7 の酸性濃度が上昇し、酸化皮膜生成に十分な酸性 (PH 3～4) 濃度を速やかに形成する。なお、前記炭酸水の酸性濃度は、使用に伴って経時的に低下するから、適時二酸化炭素を送り込み、所定の酸性濃度を維持させる。

#### 【0029】

このような状況の下で被処理部材 3 に正電流を通電すると、被処理部材 3 が前記酸性化した電解液と酸化反応し、その素地表面に不定形アルミナ ( $\text{Al}_2\text{O}_3$ ) の陽極酸化皮膜が形成される。

#### 【0030】

その際、被処理部材 3 の周囲には、水の電気分解によって酸素が発生し、陽極酸化皮膜の生成を阻害する惧れがある。しかし、前記酸素は前記バブリング状の二酸化炭素に衝突若しくは接触して移動し、前記酸化反応を維持させるから、陽極酸化皮膜が円滑かつ速やかに成長する。

#### 【0031】

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一方、このような酸化皮膜の成長に伴って、電解液である炭酸水が発熱し、その温度が上昇して良質な陽極酸化皮膜の生成を阻害する惧れがある。

しかし、実施形態では微細な気泡が大量に生成され、この気泡の移動によって浴温の放熱を促し、その昇温を防止するから、安定した酸化皮膜生成と良質な酸化皮膜を得られる。その際、このための冷却手段を省略し、若しくはその小能力化を図れるから、その分設備費の低減を図れる。

【0032】

そして、前記陽極酸化処理を所定時間実行し、十分な厚さの陽極酸化皮膜を得られたところで、二酸化炭素の供給を停止し、攪拌子10の駆動を停止して、開閉弁18を開弁する。

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【0033】

このようにすると、電解槽1内が減圧され、前記二酸化炭素の溶解度が低下し、その処理液が連通管17に導かれて貯留タンク19へ押し出され、その全量が貯留タンク19へ移動したところで、開閉弁18を開弁する。

【0034】

このため、前記貯留タンク19内の貯留液21が減圧され、二酸化炭素の溶解度が低下するため、その酸性濃度が急速に低下し、環境への実害の惧れがなくなる。そこで、開閉弁28を開弁し、前記貯留液21を排出管27から下水道へそのまま排出することができる。

【0035】

その際、貯留液19内に例えば重金属が存在する場合、前記貯留液21中から二酸化炭素が消失することで、炭酸水から分離し、前記タンク19内に沈殿するしたがって、排出管29に設けたフィルタ（図示略）を介して、他の異物や酸化皮膜と一緒に回収可能になり、前記排水の安全性を確保し、環境汚染を防止するとともに、その回収後は通常の廃棄物として処理し得る。

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【0036】

一方、本発明は前記貯留液21を再利用することができ、その場合はヒータ20を加熱し、貯留タンク19内の貯留液21を略50℃に加熱する。

このようにすると、貯留液21の炭酸水が二酸化炭素と水に分離され、これらが気液二層に分離される。つまり、気体状の二酸化炭素が上位に位置し、水が下位に位置する。

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【0037】

そこで、開閉弁25、26を開弁すれば、前記分解した二酸化炭素と水が、リターンパイプ22、23に導かれて、電解槽1および圧縮ポンプ15へ移動し、それらの再利用が可能になる。

その際、前記二酸化炭素と水は、各リターンパイプ22、23に介挿したフィルタ26、26によって、重金属や酸化皮膜、異物を除去される。

この場合、前記分解によって貯留液21から二酸化炭素が完全に抜け出るから、前記重金属や酸化皮膜等が完全に沈殿し、これらを精度良く回収できる。

【0038】

このように、本発明は水と二酸化炭素の安価な素材で、被処理部材3の陽極酸化皮膜を生成し、従来の硫酸や硝酸等による強酸性の電解液の使用を廃し、生成コストを低減するとともに、有害なガス発生下での作業環境を改善し、作業の安全性を図れ、しかも強酸性の電解液の使用を廃することで、従来の中和設備のような特別な排水設備を要せず、設備費の低減を図れる。

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【0039】

また、炭酸水の電解液に微細な気泡を大量に生成し、この気泡の移動によって浴温の放熱を促し、その昇温を防止して、陽極酸化皮膜成長時の浴温の上昇を防止し、良質な陽極酸化皮膜の生成を図れるとともに、このための冷却手段を省略し、若しくはその小能力化を図れる。

【0040】

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更に、陽極酸化処理後の処理液は、貯留タンク 19 で水と二酸化炭素に分離し、それらを電解槽 1 および圧縮ポンプ 15 に還流して再利用し、それらの有効利用と消費の節減を図るようにしたものである。

【0041】

なお、前述の実施形態では、予め前処理した被処理部材 3 を電解槽 1 に収容しているが、脱脂処理については、予め当該処理をすることなく、電解槽 1 に収容して同時に処理し得る。

すなわち、電解槽 1 に被処理部材 3 を収容後、加圧した二酸化炭素を電解槽 1 内の水 7 に送り込み、これを微粒化して高速移動させ、被処理部材 3 に衝突させることで、被処理部材 3 の表面に付着した油脂分を剥離し、脱脂させる。

【0042】

この場合、前記二酸化炭素は電解槽 1 の下方から供給され、これが水中 7 をバブリング状態で上昇するから、前記二酸化炭素が水 7 に速やかに溶解して飽和し、溶解度の上昇を促すとともに、前記攪拌子 10 と相俟って一様かつ精密な攪拌効果が得られ、前記脱脂作用を増進する。

【0043】

なお、前記方法の代わりに、前記水 7 を電解槽 1 内に霧状に噴霧し、同時に前記二酸化炭素を供給して、それらを混合すれば、それらの接触面積が更に増大して、溶解度の上昇を促すとともに、精密な攪拌効果が得られ、前記脱脂作用が一層増進する。

【0044】

図 2 および図 3 は本発明の他の実施形態を示し、前述の実施形態の構成と対応する部分に、同一の符号を用いている。

このうち、図 2 は本発明の第 2 の実施形態を示し、この実施形態は電解槽 1 の外側に電解液生成器 31 を設置し、該生成器 31 に前記ガス導管 14 と給水管 9 を接続し、該生成器 31 に導入した二酸化炭素と水とを反応して、電解液である炭酸水 32 を生成し、これを導管 33 を介して電解槽 1 へ供給している。

【0045】

図中、34 は導管 33 に介挿した開閉弁、35 は蓋体 2 に取り付けられた排気弁で、電解槽 1 の上部に設けた液面センサ 36 の検出作動によって開弁し、電解液 32 と蓋体 2 との間に滞留した二酸化炭素を外部へ排出するとともに、電解液 32 の溢出を防止可能にしている。

【0046】

すなわち、この実施形態は外部の電解液生成器 31 によって、電解液である炭酸水を生成し、これを電解槽 1 に供給することで、炭酸水生成の容易化と、炭酸水生成設備のコンパクト化と低廉化を図るようにしている。

また、前記蓋体 2 を屈曲かつ伸縮自在な蛇腹状に構成し、該蓋体 2 を介して電解槽 1 の開口部を一部を残して閉塞し、かつその先端部を電解液 32 中に没入させて、電解槽 1 の開口部の大半を閉塞している。

【0047】

そして、陽極酸化皮膜生成時は、図示のように電解槽 1 を半密閉状態にし、電解液 32 と蓋体 2 との間に滞留した二酸化炭素の流出を可及的に抑制し、一定以上滞留した二酸化炭素を前記排気弁 35 で排出し、作業の安全性と電解液 32 の溢出防止を図っている。

この場合、電解槽 1 の上部に滞留した二酸化炭素を前記電解液生成器 31 へ還流すれば、その有効利用が図れる。

また、ガス導管 14 と別個に二酸化炭素を直接電解槽 1 へ送り込めば、電解液 32 の酸性濃度を一定に維持させることができる。

【0048】

図 3 は本発明の第 3 の実施形態を示し、この実形態は電解槽 1 および貯留タンク 19 を密閉可能な耐圧構造に構成し、このうち電解槽 1 に二酸化炭素と水 7 を独自若しくは同時に導入し、それらを超臨界状態に形成可能にしている。

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## 【0049】

また、貯留タンク19は電解槽1の処理流体を一次的に貯留し、かつその処理流体を気液分離して再生し、この再生した水と二酸化炭素を電解槽1と圧縮ポンプ15へ還流させ、再利用可能にしている。

## 【0050】

前記電解槽1に脱脂前の被処理部材3を収容し、該槽1を密閉後に二酸化炭素を導入し、該二酸化炭素を圧縮ポンプ15およびヒータ12を介して超臨界状態、つまり7.4 MPaおよび31℃以上に形成し、被処理部材3を脱脂洗浄する

## 【0051】

前記脱脂洗浄後、開閉弁18を開弁し、洗浄後の二酸化炭素を貯留タンク19へ送り出し、開閉弁18を閉弁後、電解槽1に所定量の水7と二酸化炭素を導入し、これらを溶解して炭酸水を生成し、これに所定の界面活性剤を添加して、超臨界二酸化炭素のエマルジョン状態を形成する。

## 【0052】

この場合、電解槽1内が高圧状態であるから、それだけ水7に対する二酸化炭素の溶解度が上昇し、炭酸水の酸性濃度が上昇する。

そして、攪拌子10を作動して電解液を攪拌し、該電解液中に微細かつ多量の気泡を生成させるとともに、被処理部材3に正電流を通電し、前記脱脂処理した被処理部材3が電解液と酸化反応して、その素地表面に不定形アルミナ( $Al_2O_3$ )の陽極酸化皮膜が生成される。

## 【0053】

所定時間処理後、通電を停止し、かつ開閉弁18を開弁して、気液二層に分離した水7と二酸化炭素とを貯留タンク19へ送り出す。

その際、電解槽1の系内に一定の流れが発生し、これが被処理部材3を洗浄するとともに、その乾燥を促し、従来の陽極酸化処理後の水洗いを省ける。

## 【0054】

こうして生成した陽極酸化皮膜について、発明者が着色を試みたところ、着色不能であった。

これは、陽極酸化皮膜生成時に電解槽1内が7.4 MPaの高圧状態に置かれるため、陽極酸化皮膜生成と同時に封孔処理が行なわれ、陽極酸化皮膜のバルク層の孔が塞がれた結果である、と考えられる。

## 【0055】

したがって、超臨界二酸化炭素の下で陽極酸化皮膜を生成すると、封孔処理が同時に行なわれるから、従来のように封孔処理用の処理層や、該処理層への被処理部材3の煩雑な移し替えの必要がなく、設備費の低減と設備のコンパクト化並びに生産性の向上を図れる。

## 【0056】

なお、前述の実施形態は、何れも本発明を陽極酸化皮膜生成に適用しているが、これに限らず陽極酸化法と実質的に同様な原理の電解研磨に適用することも可能である。

## 【0057】

## 【発明の効果】

以上のように、請求項1の発明は、所定量の水に加圧二酸化炭素を溶解し、pH 3～4の酸性濃度の炭酸水を電解液として酸化皮膜を生成するようにしたから、従来の硫酸や硝酸のような強酸性の電解液の使用を廃し、安価かつ安全な電解液を使用して、生成コストの低減と作業環境の改善を図るとともに、特別な排水処理設備を要することなく排水でき、設備費の低減と環境汚染の防止を図ることができる。

## 【0058】

請求項2の発明は、前記被処理部材周辺の酸素に前記炭酸水の気泡を接触させて、前記酸素を移動させたから、該酸素による酸化皮膜生成の悪影響を排除し、安定した酸化皮膜生成と良質な酸化皮膜を得ることができる。

請求項3の発明は、前記炭酸水の気泡により、電解液の熱を外部へ放出させたから、従来

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のような特別な冷却手段を要することなく、酸化皮膜の成長に伴う電解液の温度上昇を防止し、良質な酸化皮膜を得ることができる。

【0059】

請求項4の発明は、前記炭酸水を攪拌して常時気泡を生成したから、被処理部材周辺に発生する酸素を排除し、安定した酸化皮膜生成を得られるとともに、酸化皮膜の成長に伴う電解液の熱の放出を促し、電解液の温度上昇を防止して、良質な酸化皮膜を得られる効果がある。

請求項5の発明は、前記被処理部材の脱脂と酸化皮膜生成を、相前後若しくは同時に処理したから、前処理工程の一部と酸化皮膜生成工程を合理的に行ない、生産性の向上を図ることができる。

【0060】

請求項6の発明は、密閉かつ加圧空間で前記酸化皮膜を生成するようにしたから、二酸化炭素の流出を防止し、その回収と再生の容易化を図れるとともに、炭酸水の酸性濃度の向上を促すことができる。

請求項7の発明は、前記酸化皮膜生成と該皮膜の封孔処理とを同時に処理したから、これらの処理工程を合理化でき、これらの処理を別々に行なう作業の煩雑と、処理槽および付帯設備をそれぞれ要する不合理と、を解消することができる

【0061】

請求項8の発明は、超臨界二酸化炭素を水に溶解し、pH 3～4の酸性濃度の炭酸水を電解液として酸化皮膜を生成するようにしたから、被処理部材の脱脂と酸化皮膜生成、および封孔処理と洗浄処理とを単一の処理槽で処理することができ、その合理化と生産性の向上を図ることができる。

【0062】

請求項9の発明は、陽極酸化皮膜生成後、前記電解液を減圧して排水するようにしたから、使用後の電解液の酸性濃度を低下し、特別な排水処理設備を要することなく、容易かつ安全な排水を実現し、同時に環境汚染を防止することができる。

請求項10の発明は、陽極酸化皮膜生成後の前記電解液を減圧かつ加熱して水と二酸化炭素に分離し、これらを排出または再利用するようにしたから、前記電解液の容易かつ安全な排水を実現できるとともに、分離後の水と二酸化炭素の有効利用を図ることができる

【0063】

請求項11の発明は、電解槽に水と加圧二酸化炭素とを導入可能に設け、前記水に加圧二酸化炭素を溶解して、pH 3～4の酸性濃度の炭酸水を生成可能に設け、該炭酸水を電解液として酸化皮膜を生成可能にしたから、従来の硫酸や硝酸のような強酸性の電解液の使用を廃し、安価かつ安全な電解液を使用して、生成コストの低減と作業環境の改善を図れるとともに、特別な排水処理設備を要することなく排水でき、設備費の低減と環境汚染の防止を図ることができる。

【図面の簡単な説明】

【図1】本発明をアルミニウム製品の陽極酸化処理に適用した実施形態を示す説明図である。

【図2】本発明の第2の実施形態を示す説明図で、電解槽の外部で電解液を生成し、これを電解槽に供給している。

【図3】本発明の第3の実施形態を示す説明図で、耐圧かつ密閉した電解槽に超臨界二酸化炭素を導入し、これに水を溶解して、陽極酸化処理している。

【符号の説明】

- |   |       |
|---|-------|
| 1 | 電解槽   |
| 3 | 被処理部材 |
| 7 | 水     |

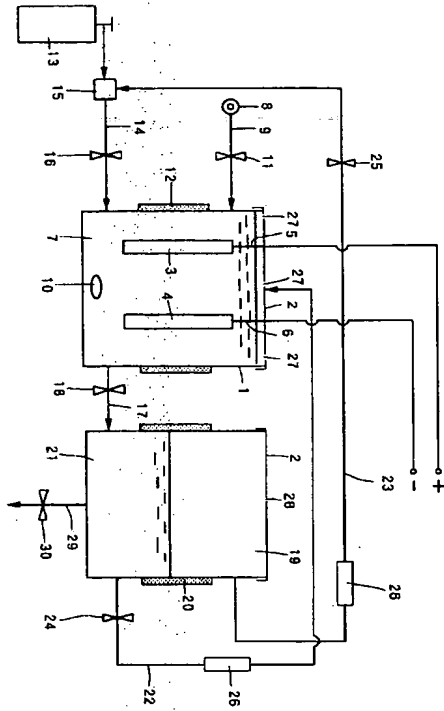
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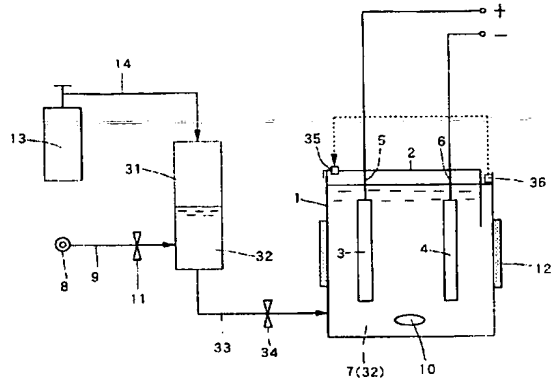
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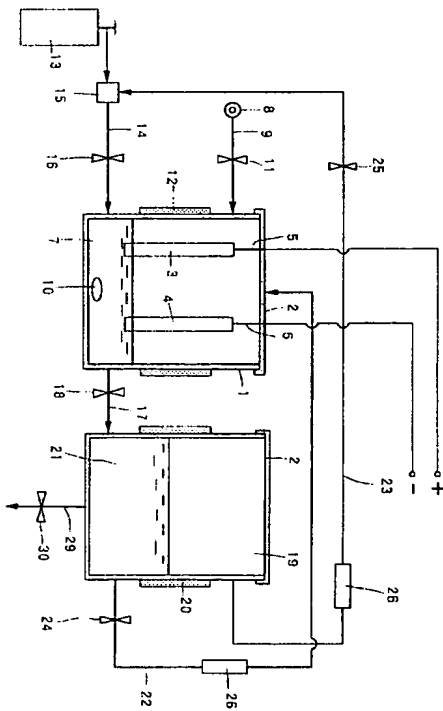
【図 1】



【図 2】



【図 3】



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フロントページの続き

(56)参考文献 特開平08-041686 (J P, A)  
特開平09-031689 (J P, A)  
特開昭61-106797 (J P, A)  
特開昭63-069999 (J P, A)  
特開昭49-000134 (J P, A)

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